CONTAMINANTS VS MICROMETEORITES FROM THE

1965 LEONID METEOR SHOWER

by

M. B. Blanchard, N. H. Farlow, and G. V. Ferry

Ames Research Center, NASA

Moffett Field, California 94035

and

H. D. Shade

Tracerlab

Richmond, California

[redacted]

SUMMARY

Particles found on collecting surfaces exposed by Ames' Luster Rocket Payload reflect a quantity of contaminants from several sources. Payload activities and particle studies were performed in a Fed Std 209, Class 100, environment. Separating collected specimens from contaminants required optical mineralogy, density, X-ray diffraction, and electron microprobe analyses.

To eliminate contaminants from future collections it was necessary to determine the effectiveness of current cleanliness control measures. Tests indicated the following were principal contaminant sources: (1) leaks in HEPA filters, (2) leaks and design deficiencies in laminar flow benches, (3) clean room garment materials and their laundering, (4) valve location relative to filter elements and materials used in fluid filtration systems, (5) cleaning and processing methods, and (6) payload design and fabrication materials.

INTRODUCTION

A rocket payload designed to collect micrometeoroids was launched into the November 1965 Leonid meteor shower from White Sands Missile Range, New Mexico, and sucessfully recovered.

An extensive contamination control program was developed for the engineering phase of this experiment and has been described by Blanchard and Farlow, 1966. Assembly, checkout, and all field operations were conducted in an environment meeting particulate requirements of Fed Std 209, Class 100. Preparation of collecting surfaces and their

[redacted]

loading into modules, which were later vacuum sealed, was performed in a similar environment. With these controls and the vacuum sealed module concept, it was felt the interior of the instrument would not be exposed to terrestrial particles larger than 0.5μ (1μ = 10^{-4} cm). Thus, collecting surfaces inside the modules should have had few contaminants larger than that size.

Preliminary results of analyses performed on particles from collecting surfaces exposed during this flight have been reported by Farlow, et al., 1966. Findings show that about one particle, 5μ and larger, was encountered for every two cm^2 of non-flight surface area. Since nearly the same concentration of particles was found on flight surfaces, questions concerning the sources of these particles arose. Further, the necessity of identifying each particle became apparent so that the few extraterrestrial particles among the contaminants could be identified.

It is the purpose of this report to describe how the sources of contaminant particles were found and eliminated through appropriate tests and modifications. Identifying major types of contaminant particles was responsible for detecting these sources.

BACKGROUND

The identification of contaminant types on collecting surfaces and the search for the sources of these particles involved two steps. First, analytical methods were used to study the physical and chemical properties of individual contaminant particles. Second, once the particles were identified, a search was made to discover how these particles escaped the control measures and reached the collecting surfaces.

Of the analytical methods employed, the optical microscope was used most extensively to delineate particle size, shape, color, texture, refractive index, and other mineralogical parameters. X-ray diffraction was used to identify compounds and minerals in conjunction with elemental composition determined by electron microprobe analysis performed on selected specimens. Particles for analysis were selected from non-flight and flight surfaces. The basic collecting surfaces were acrylic slides and vinyl films. The latter had been stripped from the modules, dissolved, and filtered onto membrane filters to facilitate microscope scanning.

The optical survey was conducted using microscopes located inside laminar flow work benches which were inside a clean room meeting Fed Std 209, Class 100 (see figure 1). The slides and filters, containing particles from the dissolved vinyl films, were stored in pre-cleaned petri dishes inside a separate laminar flow work bench (see figure 2) used only for storage. In order to perform an optical survey on a slide, it was necessary to relocate a petri dish to the microscope. The slide was then removed with a spatula and placed directly onto the microscope stage. Therefore, during handling the slides were never touched except by the spatula. When a survey had been completed the slide was returned to the petri dish which was then relocated to the laminar flow storage bench.

It was possible to characterize contaminants with high confidence since each microscope possessed the capability to examine a particle using several types of illumination without removing the particle from the field of view. In transmitted light: bright field, dark field, phase contrast, and polarized light were used. In incident light:

A-37406-9

Figure 1

A-37406-11

Figure 2

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
AMES RESEARCH CENTER, MOFFETT FIELD, CALIFORNIA

bright field, dark field, and polarized light were used. The entire surface of each non-flight slide was surveyed at 100x. Contaminants were counted, identified, and photomicrographed at 400x. Using these photomicrographs, a contaminant compendium was prepared based on morphology of the particles. Several variations of a given contaminant type were selected to complete the compendium. Identifications revealed the following to be principal contaminants: skin cells, acrylic plastic shreads, glass chips, metal chips and shreads, pieces of RTV (silicone rubber sealant), cloth fibers, and bacteria. These are illustrated in figure 3.

Identification of the contaminant types points to several contaminant sources. One was the flight payload. Using X-ray diffraction, one specimen was identified as a certain type of bronze. The source in the payload was a sintered bronze, oil impregnated, bushing located in an area remote from the modules. Another source was the modules themselves. Analyses using the electron microprobe identified a group of opaque particles found on flight slides to be shreads of metal plating. These metal shreads were generated by screws being threaded into tapped holes on the modules during installation on the flight instrument. But major sources were also found within the laboratory. Slide washing procedures appeared to be less effective than they should have been considering the specifications of the equipment being used. Laminar flow facilities were not excluding particles they were designed to eliminate. Clean garments were producing contaminants which should have been removed by proper laundering. Therefore, extensive tests were conducted to determine the limitations of the clean

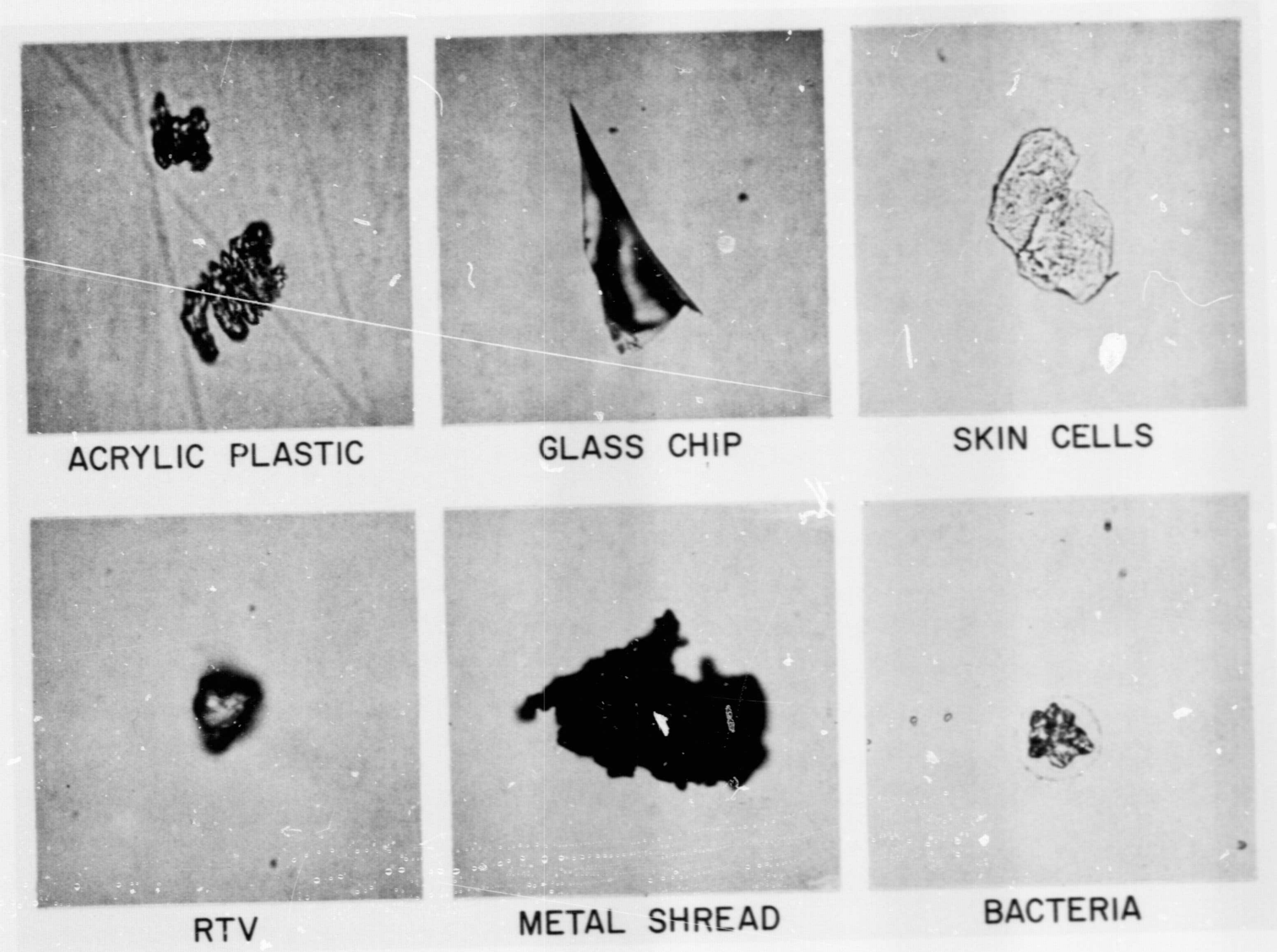

ACRYLIC PLASTIC
GLASS CHIP
SKIN CELLS
RTV
METAL SHREAD
BACTERIA

Figure 3

room equipment and procedures. Then, modifications were made to the equipment, and methods, so that contaminants could be eliminated from future experiments.

TEST RESULTS AND MODIFICATIONS

Every piece of clean room equipment was tested by appropriate ASTM or Fed Std 209 test methods to determine the effectiveness of the control equipment. Procedures being used by clean room personnel were reviewed to detect inadequacies in operating methods. In almost every case examined, deficiencies were detected.

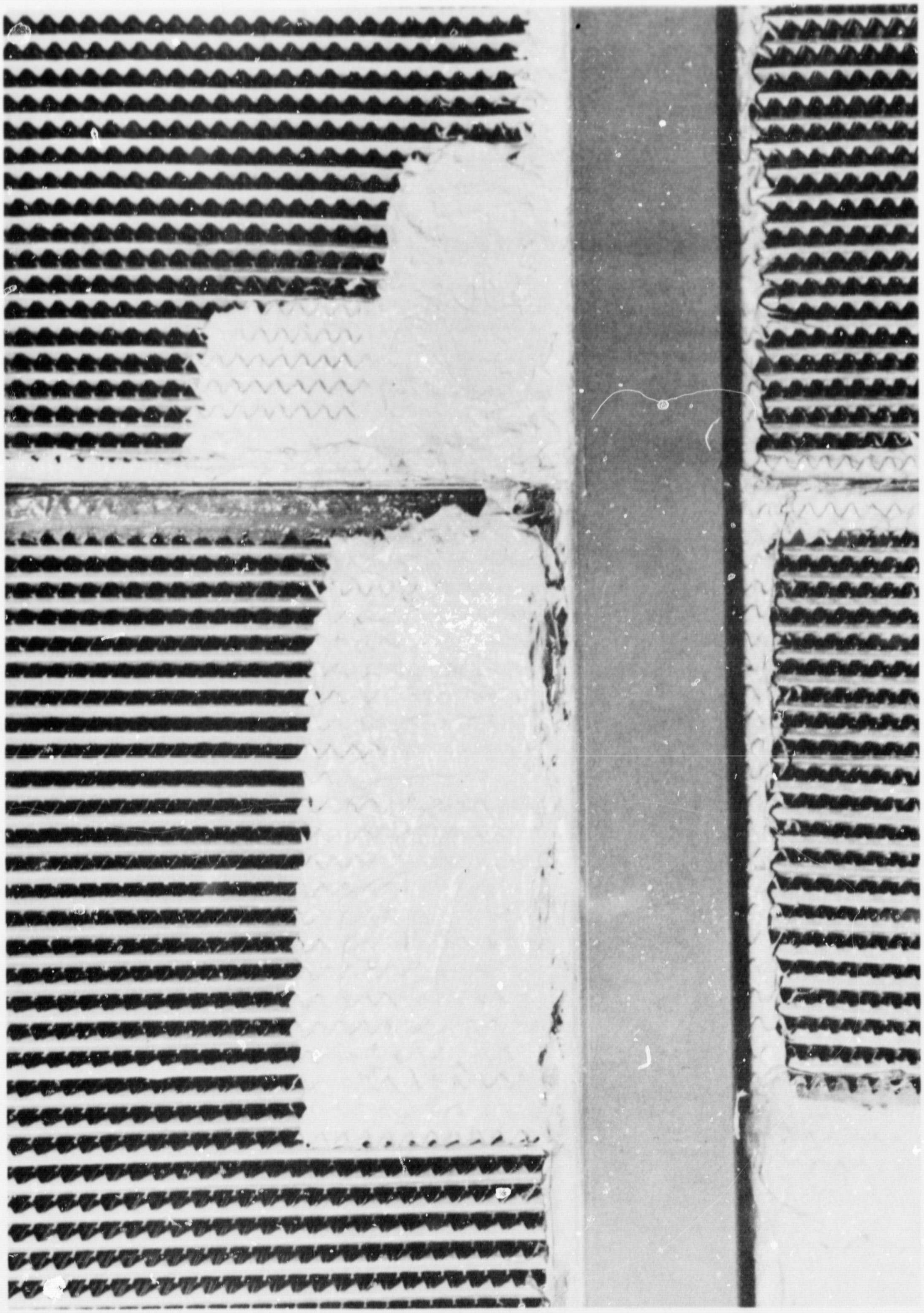
Clean Room Filters - Air inlet filters being used for the three clean rooms at Ames were nearly new. Each had a honeycomb-type structure with a plywood frame. The manufacturer's data indicated they had passed a 99.97% filtration efficiency test for 0.3μ DOP particles. Tests were performed on each of these filters, while installed and operating, by scanning the entire filter face using a light scattering aerosol photometer and a Model II (Echols and Young, 1963) smoke generator according to ASTM F50-65T. Immediately upstream of the filter, counts for particles 0.5μ, and larger, averaged 40,344/liter, while downstream the counts were 4,108/liter, indicating about 90% filtration efficiency. Application of RTV silicone rubber sealant to detected holes and leaks corrected the problem.

Laminar Flow Work Stations - Next, attention was directed to the six laminar flow work benches and one laminar flow fume hood inside the clean rooms. All of these filters possessed pleated media with aluminum separators and steel frames. One hundred percent of each filter was scanned and location maps were made of leaks. Leaks were

plugged using RTV sealant and then retested. Figure 4 shows results of RTV plugging performed in one area on a filter and a completed laminar flow bench is shown in figure 5. At the point where two 3-feet-long filters were joined in one frame to make a continuous filter 6 feet long, considerable leakage was always detected. As can be seen, considerable leakage occurred at all filter frame/cabinet interfaces. Designs used for maintaining a seal at this location were unusually poor.

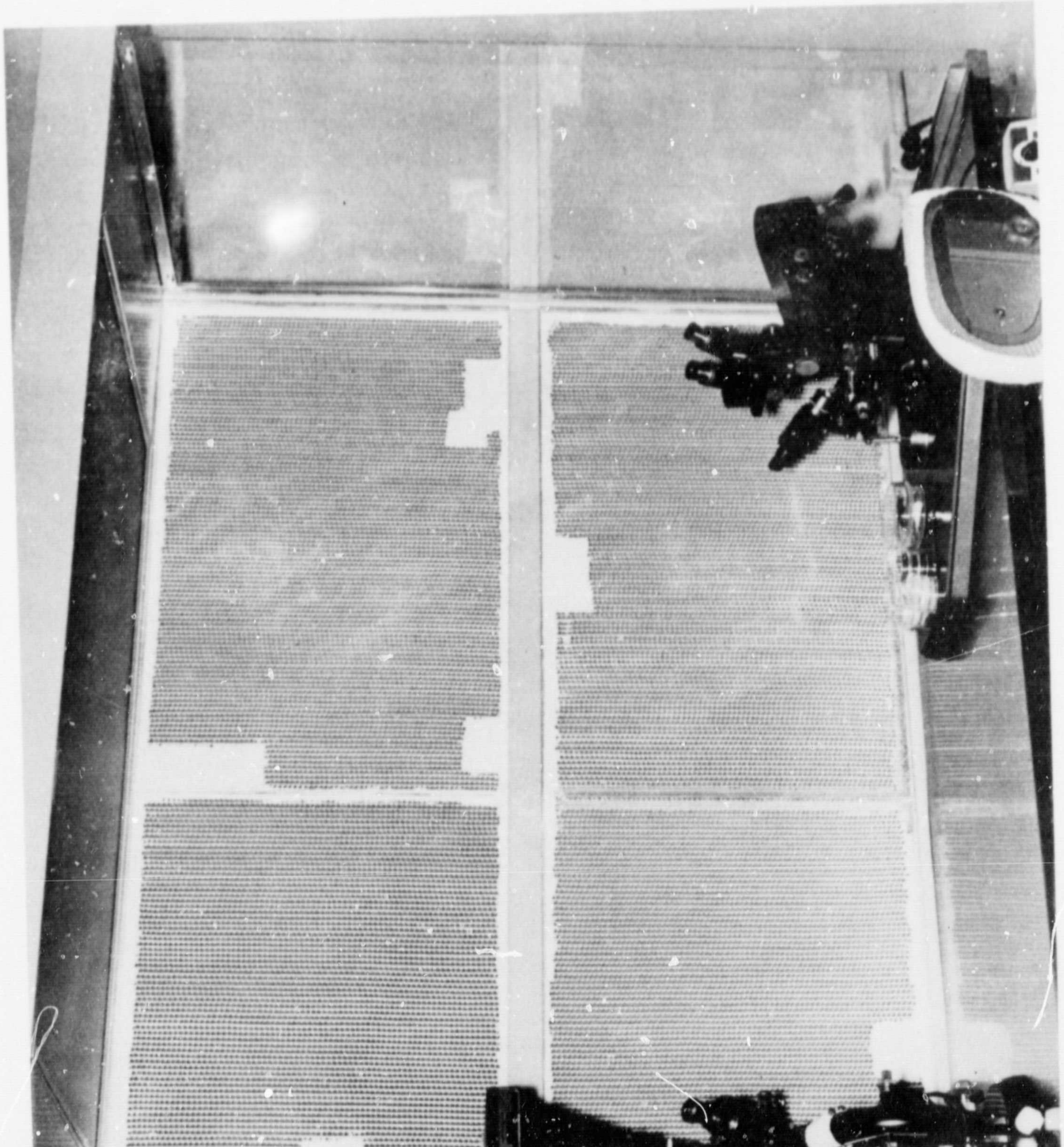
Prior to testing filters in the cabinets the protective grills had to be removed. The side of each grill facing the filter was found to be greasy and dirty (figure 6). Threads were observed to be working their way out of one filter. An assemblage of dirt particles were found deposited immediately in front of the filter on the frame (figure 7), and in one instance an unused screw hole demonstrated its contribution to particulate contaminants (figure 8). Each cabinet housing was characterized by many leaks at joints, seams, and electrical outlets. Discarding protective grills and sealing cabinet holes with clean vinyl tape corrected the problem. Polyethylene-covered fiber glass sound proofing located inside of the blower-motor chamber had torn loose and was abrading inside the blower squirrel cage on one cabinet. Removal of the sound proofing was performed, but some sources remained for glass particles to be transported directly into the plenum (figure 9).

<u>Clean Room Garments</u> - At one stage during the optical survey a large quantity of similar particles predominately 25μ in size were detected on the slides being examined. With each work shift the



A-37533-6

Figure 4



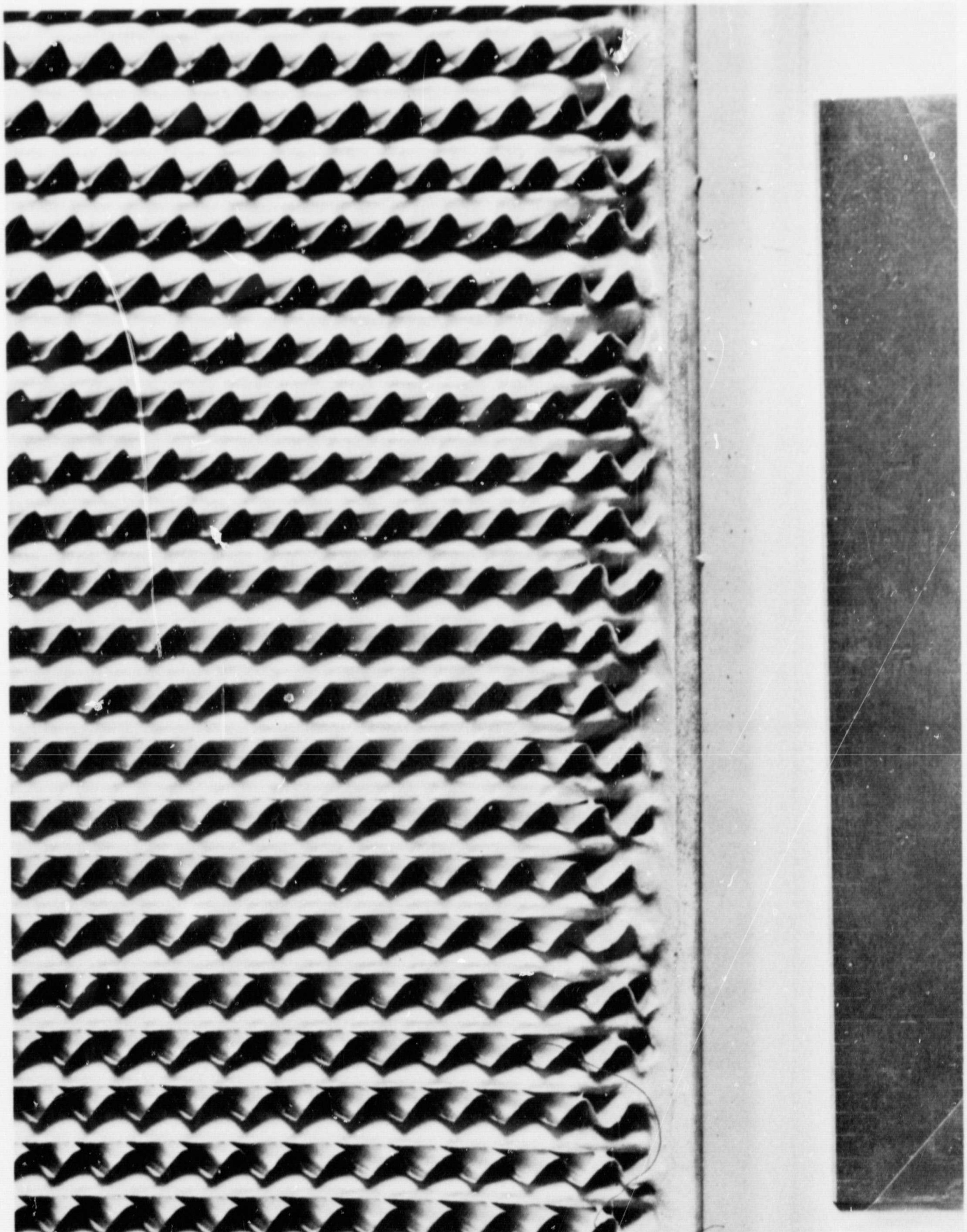
A-37533-1

Figure 5

Figure 6

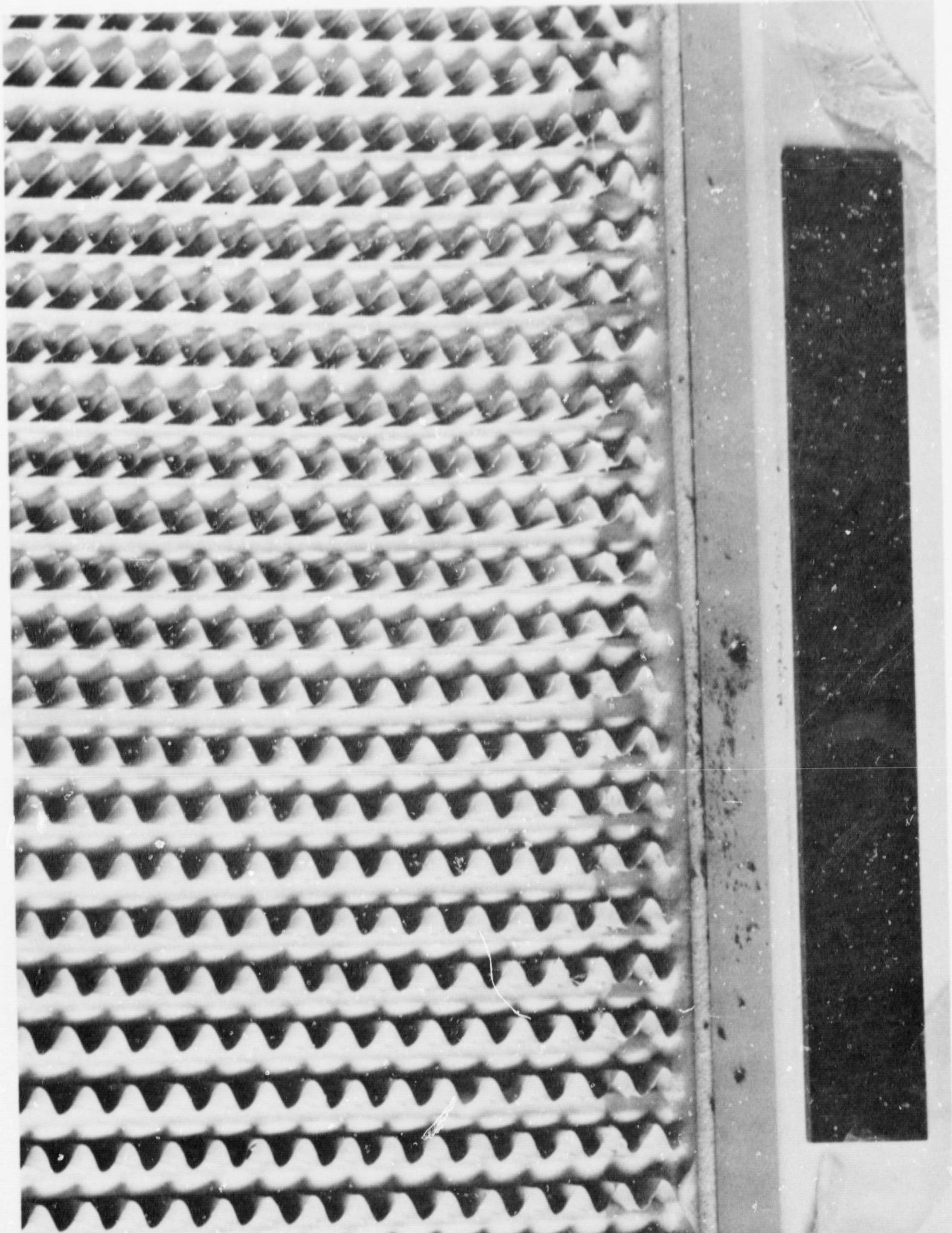
A-37427

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
AMES RESEARCH CENTER, MOFFETT FIELD, CALIFORNIA



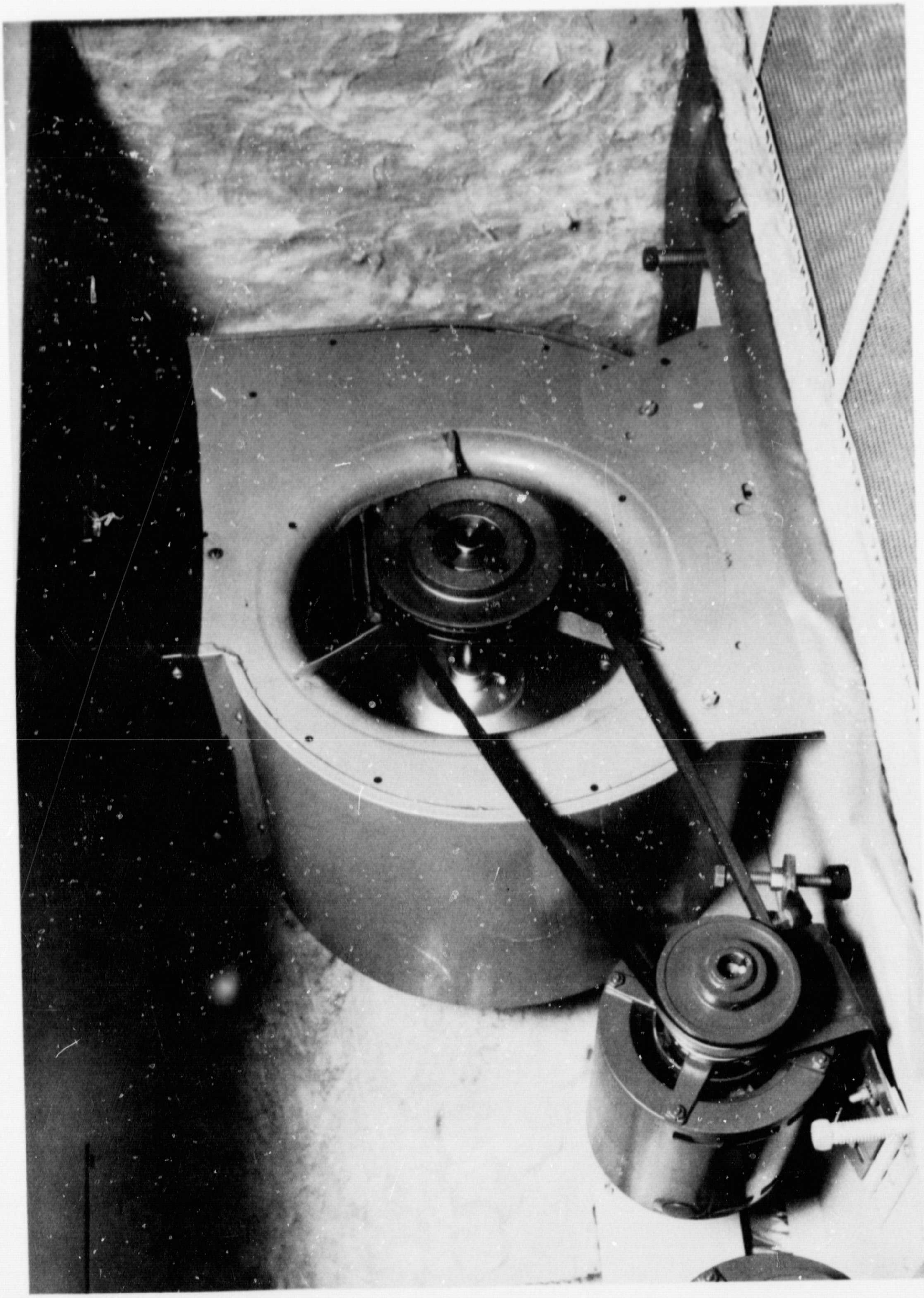
A-37426

Figure 7



A-37429

Figure 8



A-37430

Figure 9

quantity detected increased and a complete operational shut down was necessary. Tests showed these particles (figure 10) were being generated from the clean garments. They were a combination of soap and salt particles originating from the laundering service and were present in all laundered garments. Changing to a different laundry service solved this problem.

Gloves used by the microscopists included three types: (a) continuous filament stretch nylon, (b) monofilament nylon tricot, and (c) monofilament nylon tricot having the front panel impregnated with plastic. Types (b) and (c) had been highly recommended by the manufacturer for Class 100 use. Tests showed the monofilament nylon to be rather brittle. Large quantities of nylon filaments (figure 11) were periodically breaking away and falling onto the slides and microscope stages, introducing serious contamination during microscope scanning. Types (b) and (c) were both poor from this standpoint. Results indicated type (a) to be superior from a particle generation aspect and to yield fewer contaminants during use. Therefore, this type has been adopted for exclusive use in all clean room activities at Ames. Table 1 is a comparison of particle counts obtained on garments from two separate laundry services, with Class B requirements of ASTM-F51-65T. Note that only an occasional garment met the cleanliness level specified for Class B even though the laundry supplied certification statements indicating all met these requirements.

<u>Fluid Filtration Systems</u> - Attention was directed to the fluid filtration systems, since nearly everything used in the experiment and analysis was subjected to cleaning processes using filtered liquids.

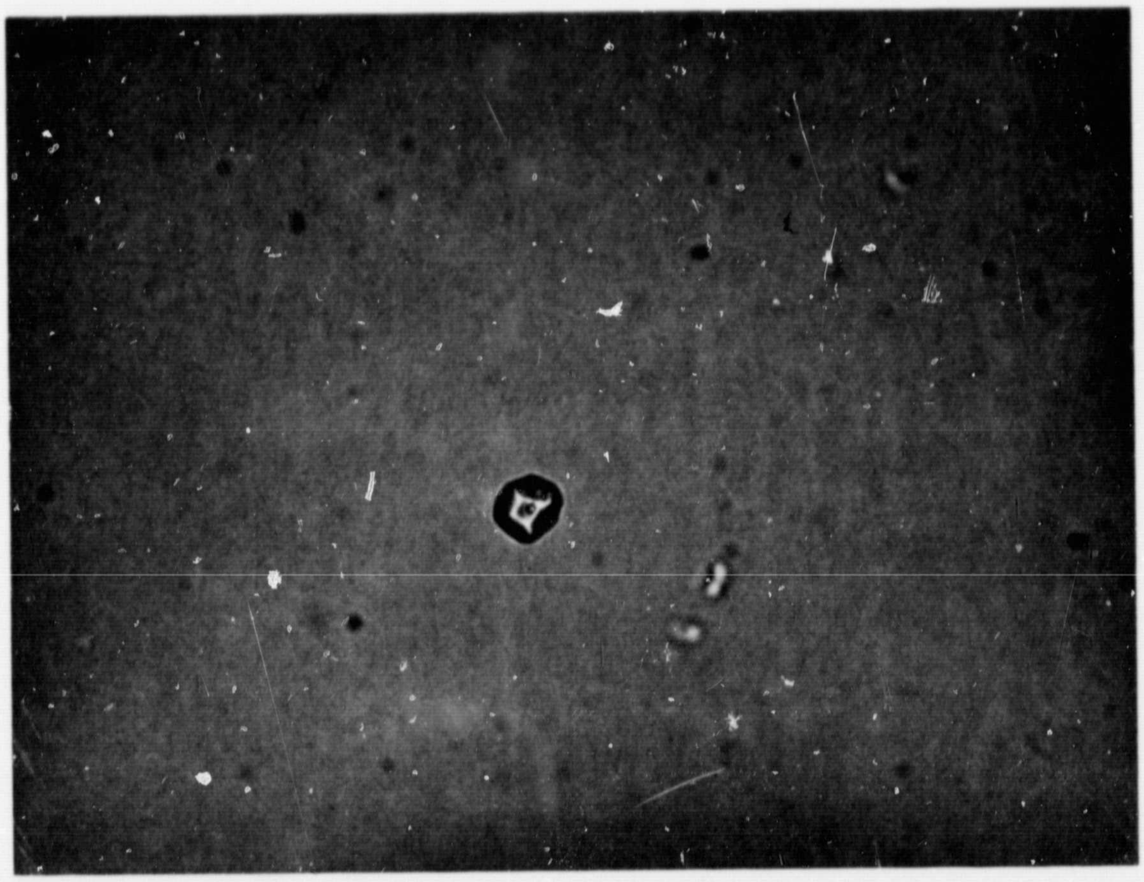
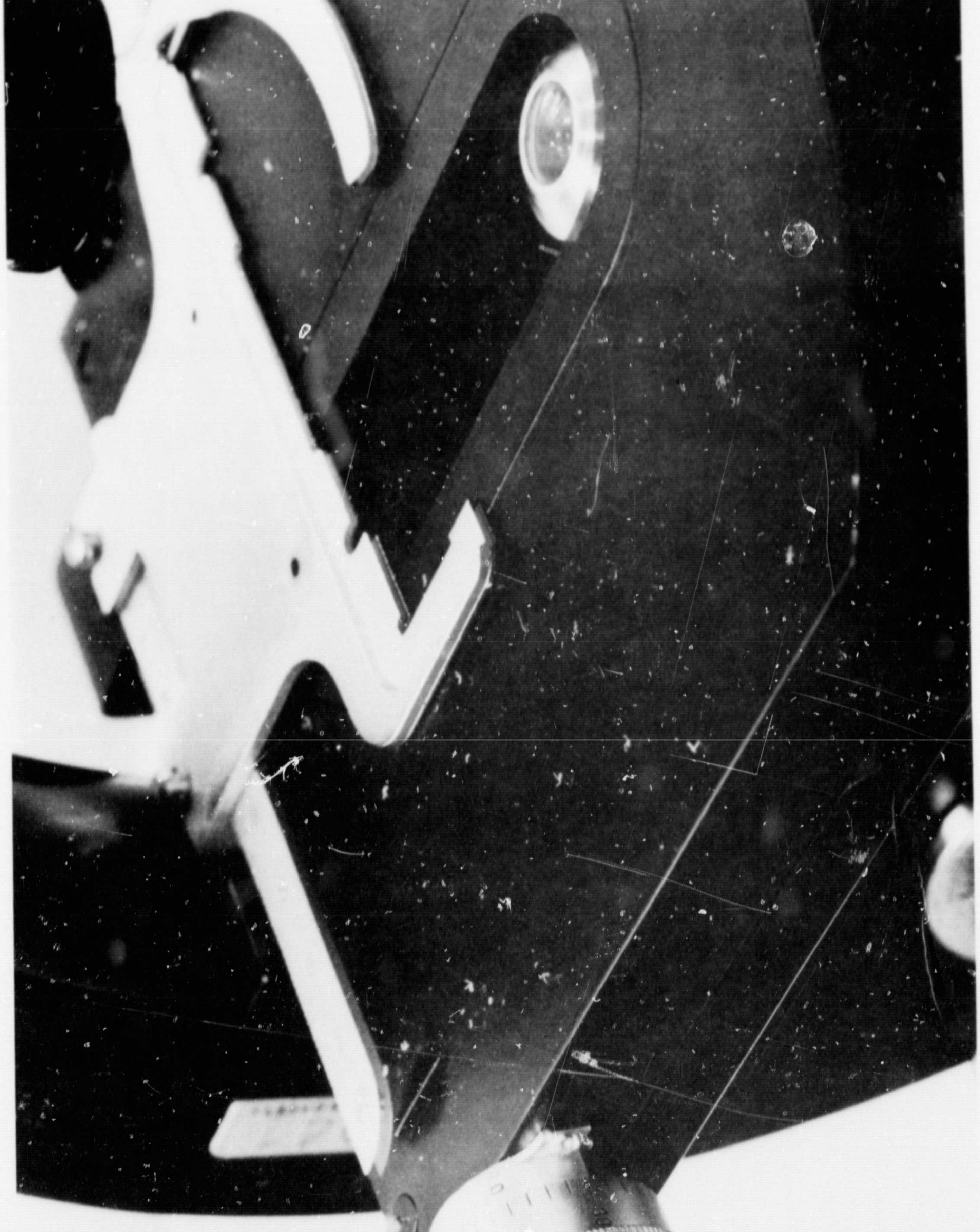


Figure 10



A-38106

Figure 11

TABLE 1

TESTS PERFORMED ON CLEAN LAUNDERED GARMENTS

	No. of particles 5μ and larger/sq ft of garment surface		
	ASTM-F51-65T		
Garment	Class B requirements	Laundry B	Laundry A
Hat	< 5000	8,260	19,160
			49,860
Shoe covers	< 5000	14,460	10,260
			87,160
Coveralls	< 5000	7,660	2,330
		2,670	9,980
		5,220	41,760
		6,700	54,660
		20,310	

Tests conducted in accordance with ASTM-F51-65T.

All particle counts have been corrected for background.

The necessity to filter all cleaning fluids prior to use was graphically illustrated by a series of tests. A new 5 gallon can of trichlorotrifluoroethane was selected at random, and a one-liter sample was passed through a membrane filter with a 0.8μ pore size. Results are shown in figure 12. This is probably the worst contamination encountered and certainly is far from the manufacturer's specifications. However, it shows the burden and importance placed on filtration systems used in clean room activities, and emphasizes the necessity of fluid filtration.

Fluid filtration has generally been performed using only one filter in the system (single filter concept). A test of the efficiency for this approach was performed by positioning three filters in series using filter holders recommended by the filter manufacturer. Figure 13 represents the quantity of contaminants found on the 25-mm filter positioned first in line; figure 14 shows the same for the filter positioned second in line; figure 15, the same for the final filter. All represent contaminants from a one-liter test sample of trichlorotrifluoroethane. Figure 16 reflects the concentration of contaminants found in the one-liter sample after having been filtered through the previously mentioned filters. This result was obtained by taking the output from the third filter and filtering it again, using a vacuum filtering test apparatus, through a 47 mm, 0.8μ pore size, membrane filter. These results demonstrate the single filtration concept to be inadequate.

During testing of several filtration system configurations, it was discovered that the location of the metering valve, in relation to the filter, was vitally important. Tests were performed on three

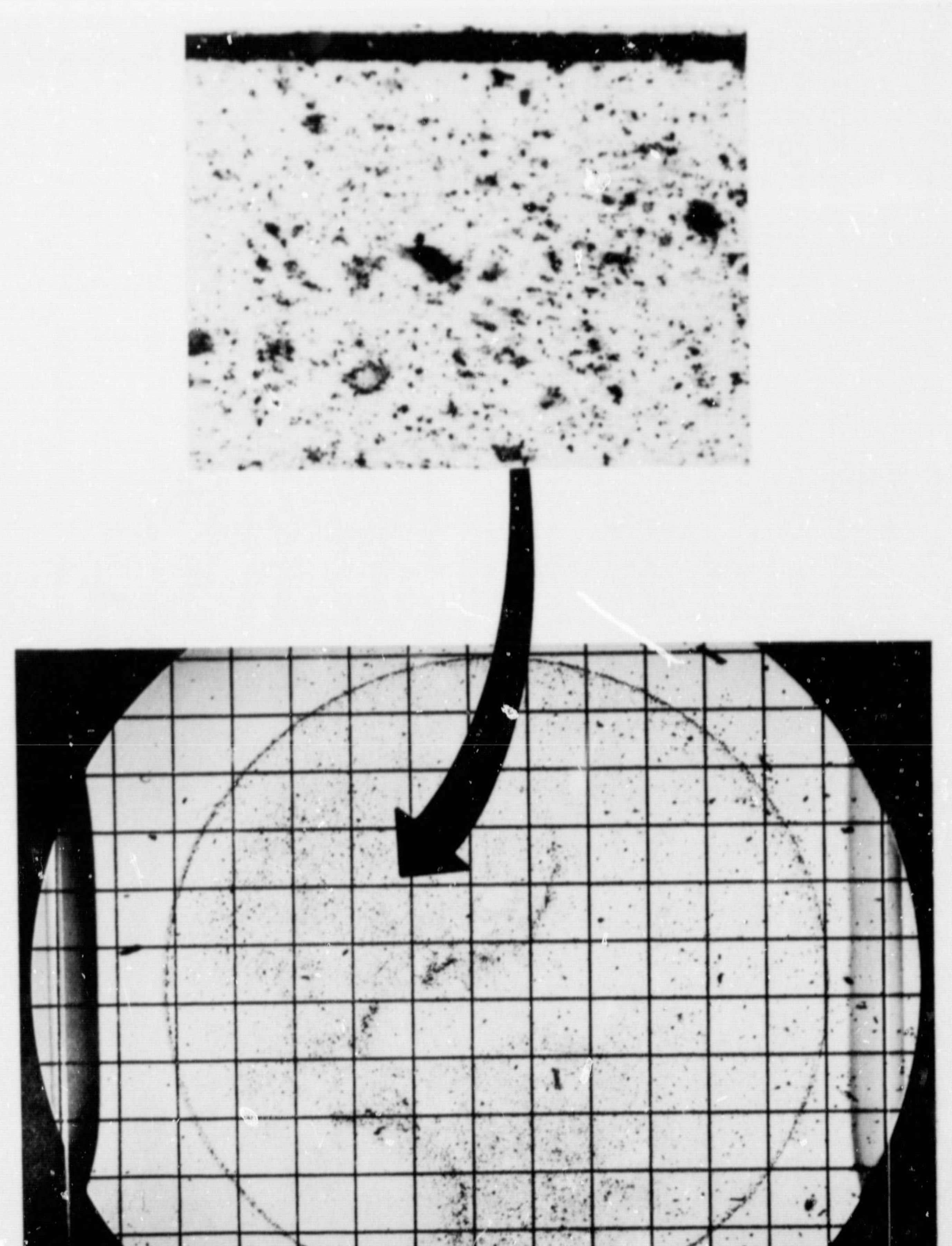


Figure 12

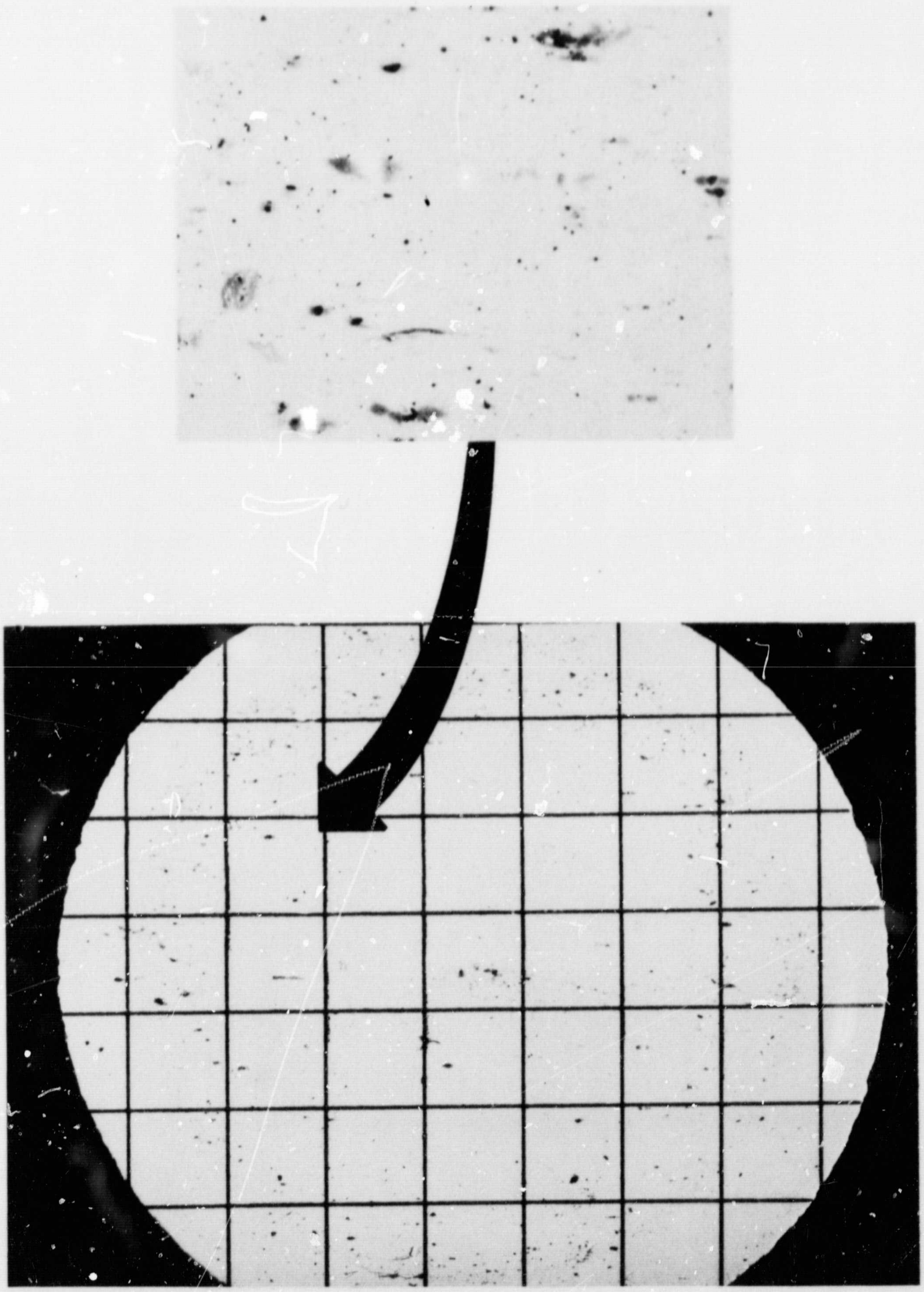


Figure 13

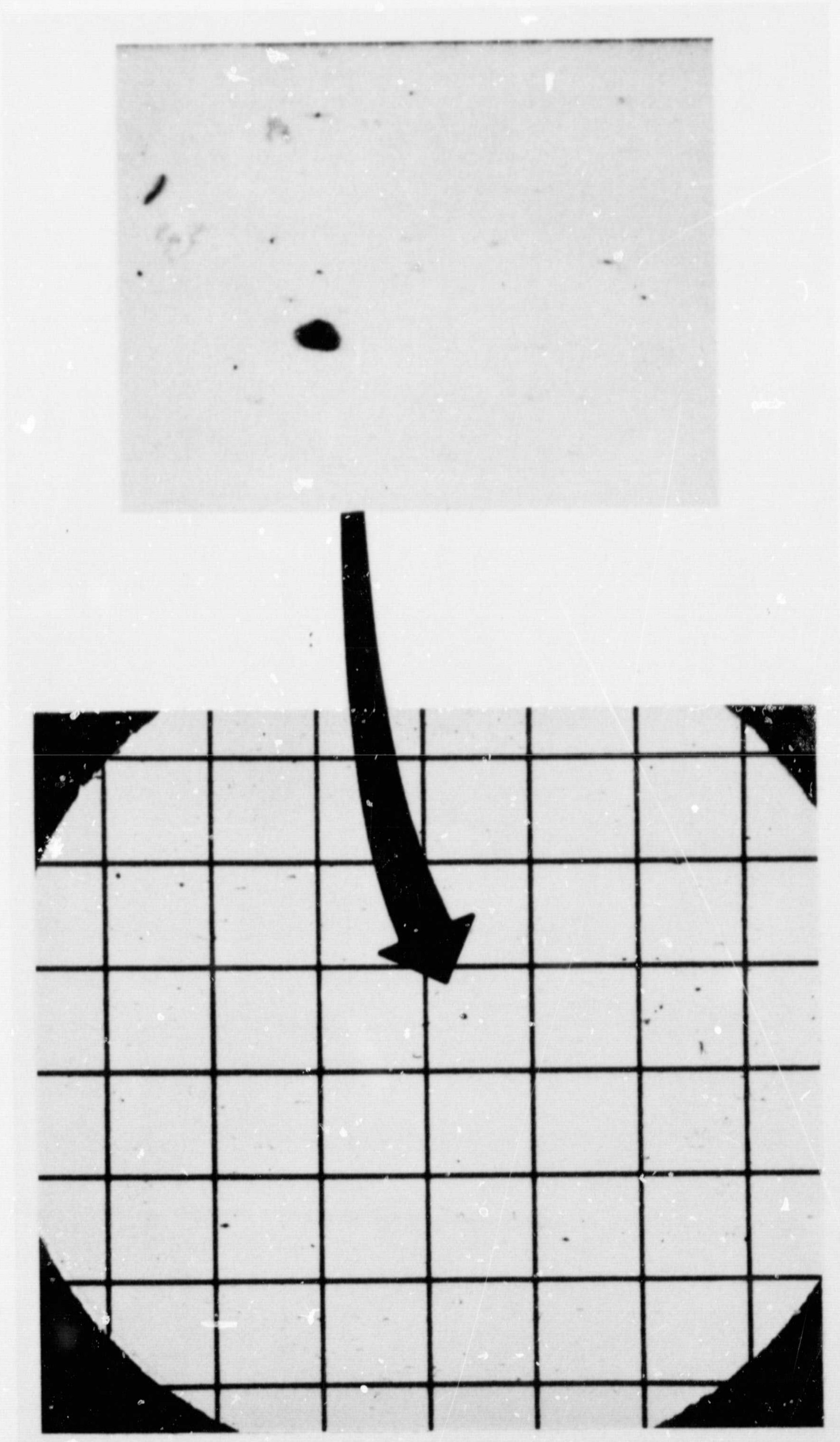


Figure 14

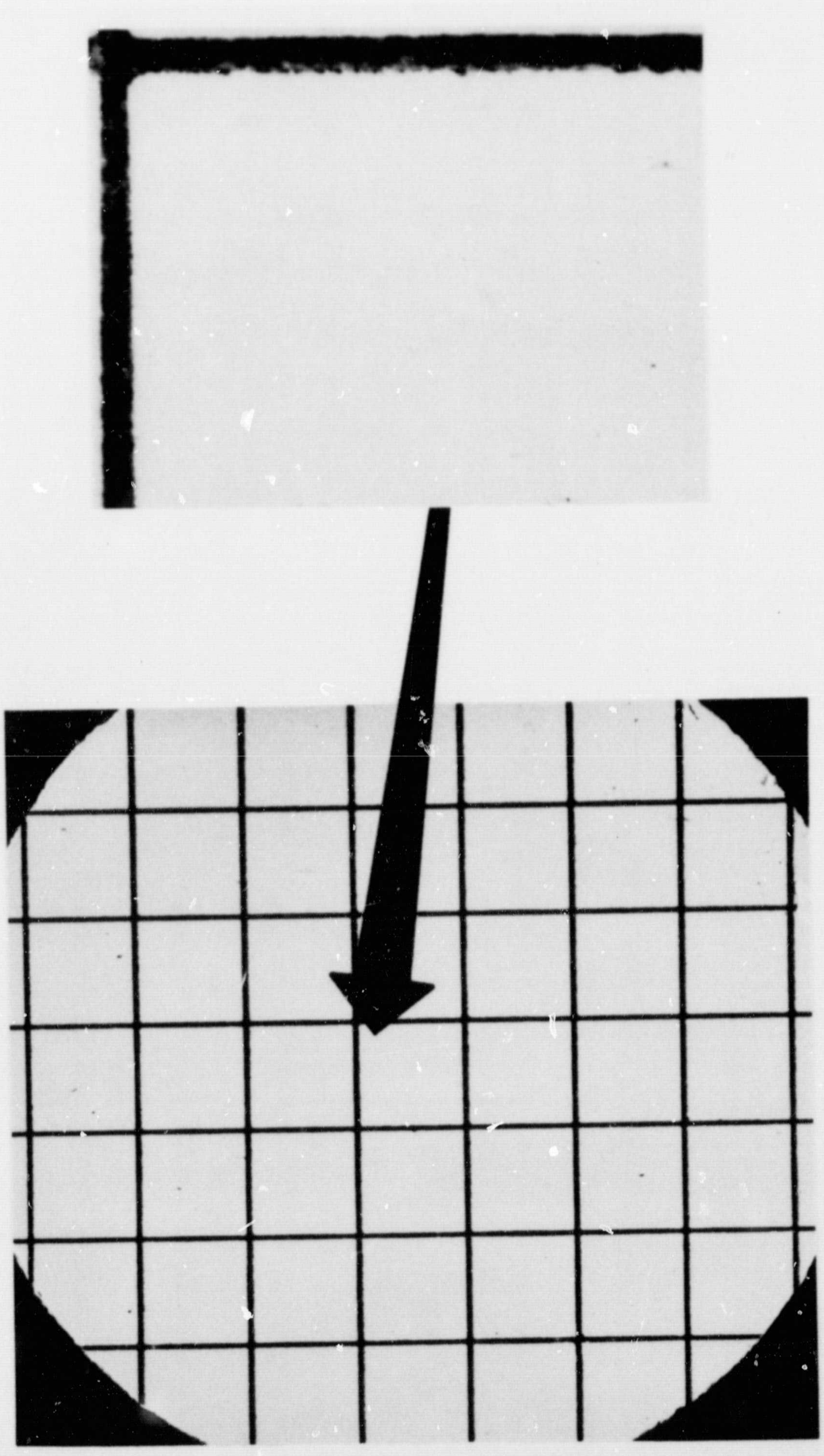


Figure 15

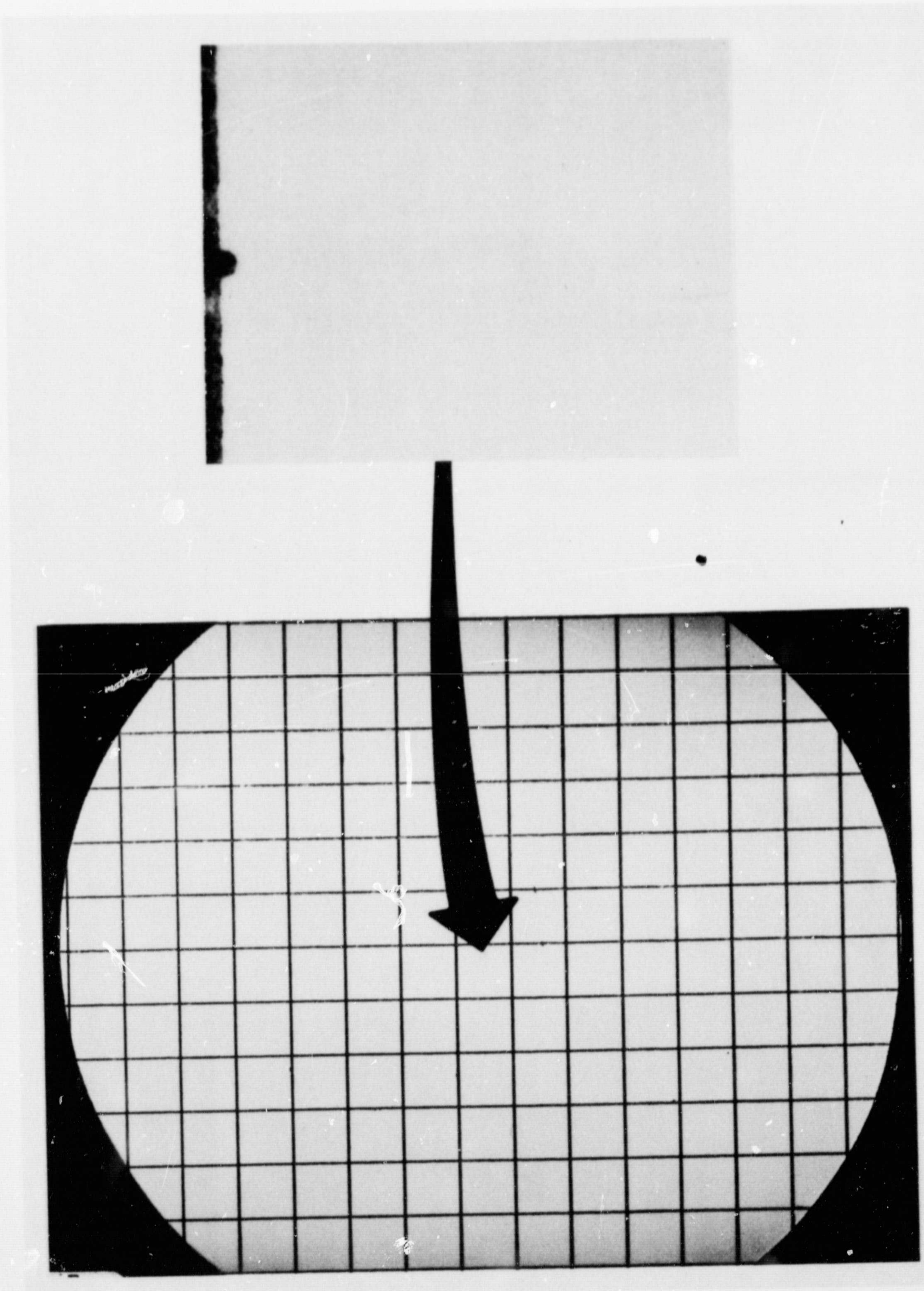


Figure 16

configurations. All utilized stainless steel pressure vessels and membrane filters. Configuration "A" possessed two 6-inch membrane filter holders in series located between the tank and outlet valve. Configuration "B" possessed three separate 25-mm filter holders in series and had a valve positioned between the second and third filter. Configuration "C" possessed three 25-mm filters in series in one filter holder and a valve positioned between the tank and the first filter. After each system had been cleaned, flushed, and had new filters installed, a gallon (or more) of fluid output was filtered through a 47-mm membrane filter (0.8μ pore size) using a vacuum filtering test apparatus. Results of particle counts, along with a schematic illustration for each configuration, are given in figure 17. Counts on filters placed on both sides of the valve in configuration "B" showed the valve increased the number of contaminants by a factor of 10, demonstrating there is little value in filtering fluids upstream of a valve. Counts on three filters in series from configuration "C" located downstream of the valve reflected a non-linear decrease in the number of contaminants found of each successive filter, demonstrating one filter after a valve is clearly insufficient. Repeated samplings from configuration "C" yielded an output concentration ranging from 29 to 42 particles (larger than 5μ) per liter. This represents the cleanest fluid level that can be expected from configuration "C." The particular valve used in this configuration had been carefully selected and considered to be one of the least contaminant generating available commercially. It had a stainless steel body, Teflon tipped stem, Viton-A "O" ring, and a nylon gland

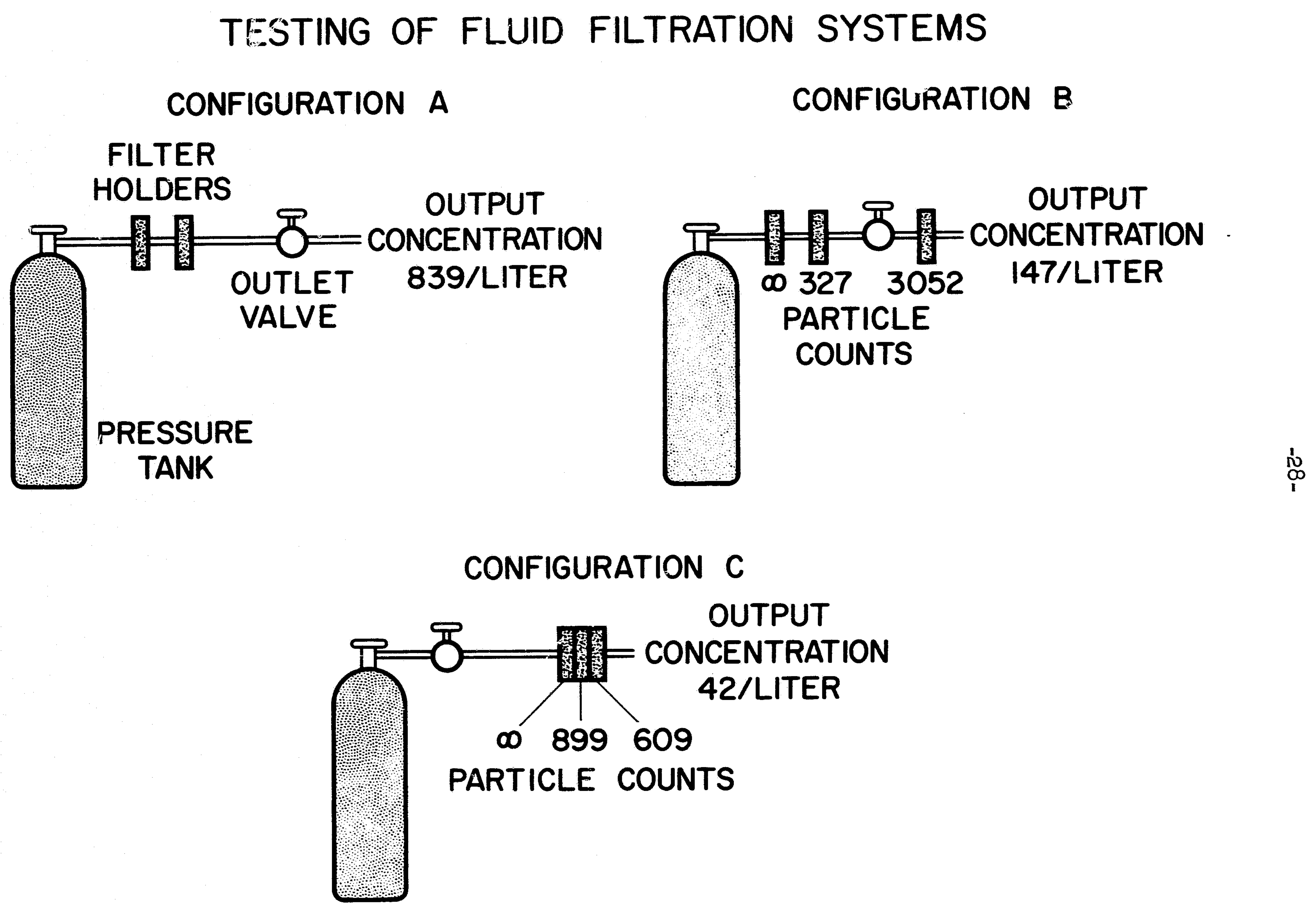

TESTING OF FLUID FILTRATION SYSTEMS
CONFIGURATION A
FILTER HOLDERS
OUTLET VALVE
OUTPUT CONCENTRATION 839/LITER
PRESSURE TANK
CONFIGURATION B
∞ 327 3052
PARTICLE COUNTS
OUTPUT CONCENTRATION 147/LITER
CONFIGURATION C
OUTPUT CONCENTRATION 42/LITER
∞ 899 609
PARTICLE COUNTS

Figure 17

(top and bottom) with a nylon handle. Figure 18 illustrates the form of configuration "C" which has now been adopted for use at Ames in cleaning activities.

A commercially available pressure rinser, employing terminal filtration, was also tested. Prior to testing it was thought that this unit could be modified to configuration "C" by employing three filters in the single filter holder. The rinser was thoroughly cleaned and flushed with triple filtered output from the test unit of configuration "C." A single filter was installed and new triple filtered fluid (from the test unit of configuration "C") was then cycled four times through the rinser. The concentration of contaminants shown in figure 19, caught by the filter in the pressure rinser, was unexpectedly high. Optical examination of the 25-mm membrane filter (0.8μ pore size) indicated essentially all material came from 3 sources within the pressure rinser. These sources were rubber (no. 1), brass (no. 2), and a gray metal coating on the spring (no. 3), as shown in figure 20. Thus, the pressure rinser tests emphasized a fundamental design requirement for any fluid filtration system. The operation of the filtration system must not be contaminant generating. Design of the metering valve used in the pressure rinser does not meet this requirement.

Filter support screens were found to be an important source of metal shreads occurring in outlets from fluid systems. This was due to the periodic breaking away of pieces from the wire mesh (figure 21). Chemically etched screens are superior from this standpoint. A constant inspection and test program has been necessary to detect equipment deterioration such as this and to prevent these contaminants from getting onto the collecting surfaces during initial cleaning.

A-37749-7

Figure 18

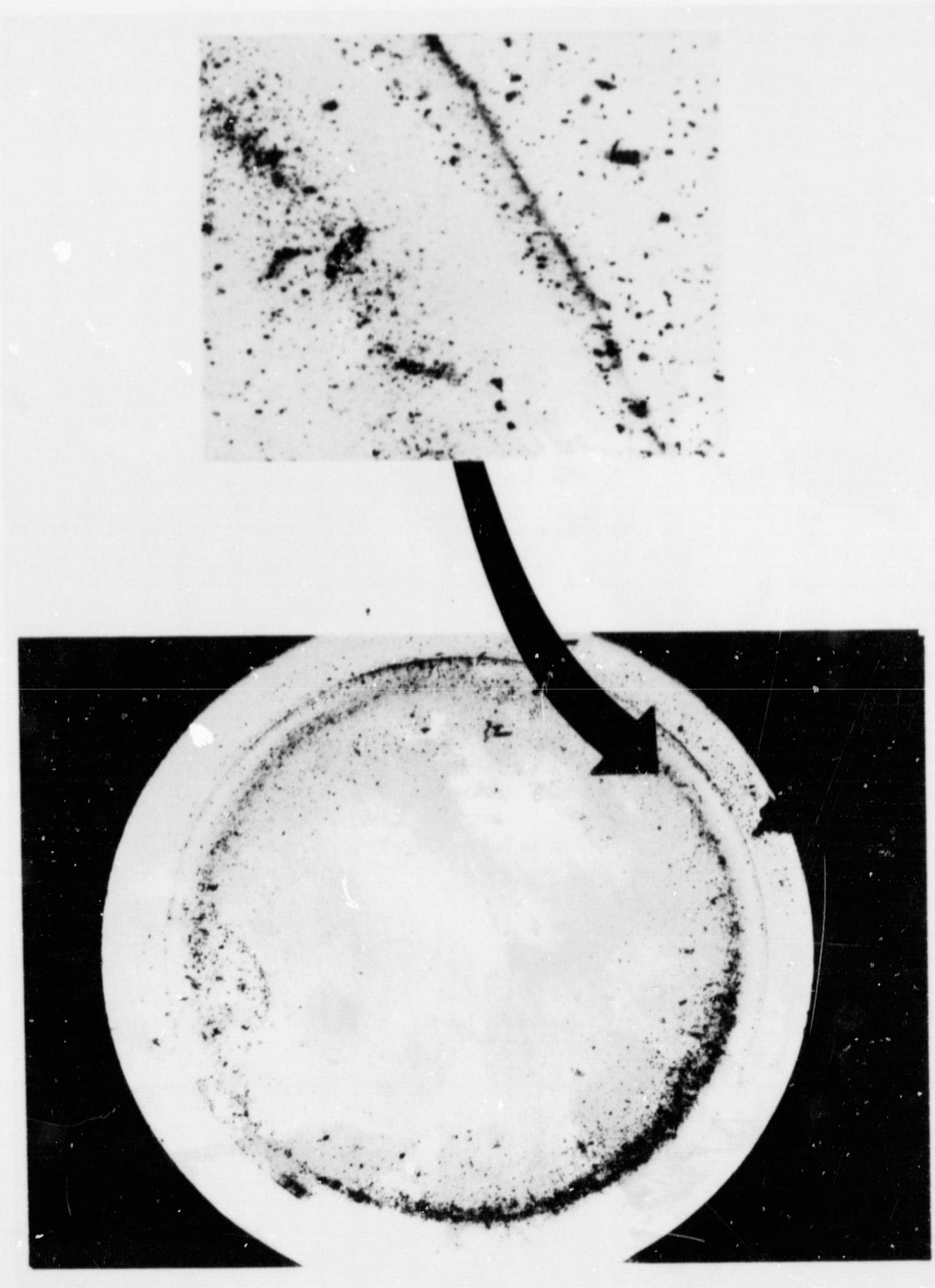
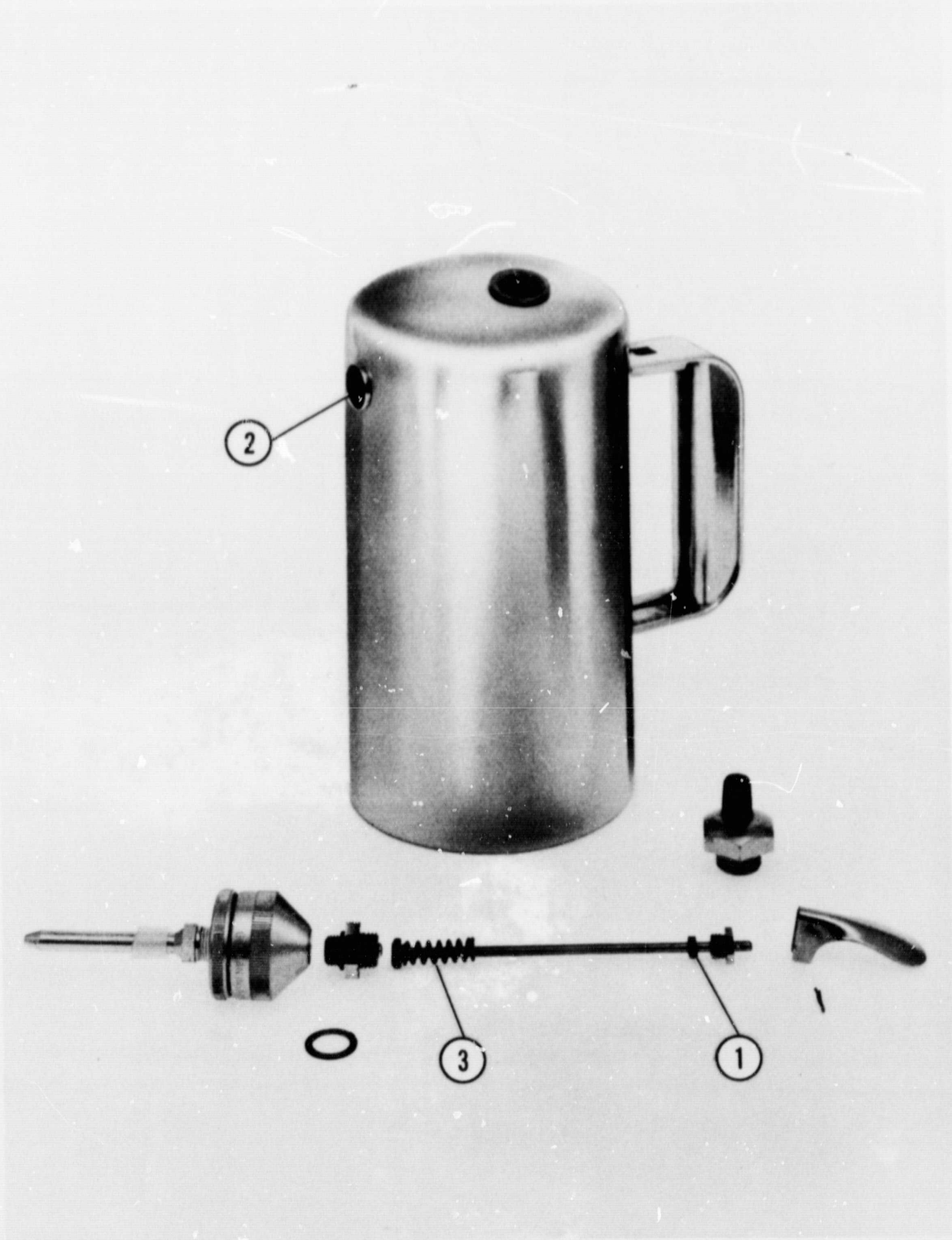
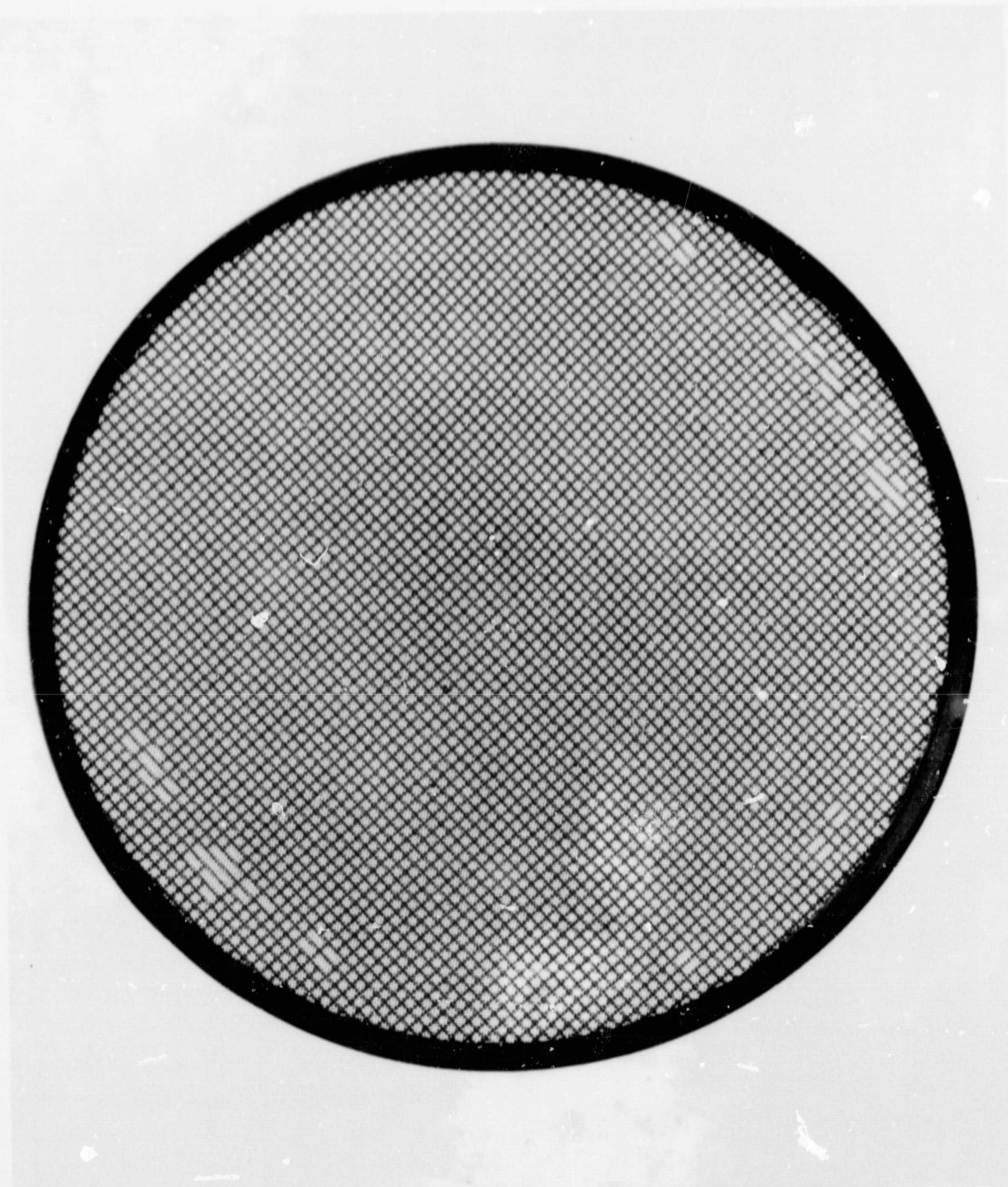


Figure 19



A-37749-11.1

Figure 20



A-38104

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
AMES RESEARCH CENTER, MOFFETT FIELD, CALIFORNIA

Figure 21

Handling and Processing Methods - Once test results demonstrated that it is possible to design an effective fluid filtration system, the cleaning and processing of parts was examined. Parts for the 1965 flight instrument had been cleaned to Level I, of 10M01671 A MSFC. A series of cleaning and processing tests was performed using configuration "C" (figure 18) as the fluid filtration system. Tests were conducted using samples made from sheet metal with riveted and machined parts attached. Results indicated that it was possible to reduce the contaminant level for future experiments considerably below that which had previously been obtained. Tests showed that a maximum contamination of less than 500 particles (5-100μ size range) for every 0.09 m^2 (1 ft^2) of surface area rinsed could be expected. Among these, no more than 10% would be metal.

Skin cells and bacteria were being generated by personnel cleaning the slides and by microscopists performing the optical survey. The quantity of skin cells was markedly reduced by requiring each person to thoroughly wash his hands and face prior to starting or returning to an activity after an interruption. Bacteria counts were markedly reduced by eliminating talking activities directly over the microscope. Once again, this pointed to the necessity of constantly reviewing methods and emphasizing the importance of continual testing at each step in the sequence.

DISCUSSION

In spite of an extensive contamination control program some contaminants remained on the collecting surfaces during the rocket

flight into the Leonid shower. About one contaminant particle, 5μ and larger, per two cm^2 was found. These particles gained access to the surfaces via three important avenues:

First, the commercially available contamination control equipment and materials used did not provide the degree of cleanliness anticipated due to leaks and design deficiencies. When such equipment was used without testing and modification, it contaminated the materials which it was attempting to protect.

Second, activities performed by personnel in the clean rooms often unknowingly produced contamination problems. On occasion, procedures adopted to prevent one type of contaminant introduced a new one.

Third, the actual flight instrument generated contaminants during checkout and operation in flight. The sources for these contaminants were, again, design deficiencies and field activities performed by personnel.

Consequently, it has become clear that all phases of any contamination control program must be constantly reviewed. A continuous monitoring program is necessary in order to assess the cleanliness level at any one time in the program. Merely conducting activities in a Fed Std 209, Class 100, environment in accordance with the specifications' guidelines is very clearly inadequate.

REFERENCES

[1]Contamination Control During Design, Fabrications, Test, and Launch of an Upper Atmospheric Rocket Payload, M. B. Blanchard and N. H. Farlow, Contam. Contr., 1966, Vol. 5, pp. 22-25.

[2]Sampling With a Sounding Rocket During a Leonid Meteor Shower, N. H. Farlow, M. B. Blanchard, and G. V. Ferry, J. Geophys. Res., 1966, Vol. 71, pp. 5689-5693.

[3]Studies of Potable Air Operated Aerosol Generators, W. H. Echols and J. A. Young, NRL Rept. 5929, Washington, D. C., 1963.